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IS 6712 (2003): o-Dichlorobenzene [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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(पहला पुनरीक्षण)

Indian Standard

o-DICHLOROBENZENE — SPECIFICATION

(First Revision)

ICS 71.080.20

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 2003 **Price Group 3**

Organic Chemicals (Miscellaneous) Including Aromatic Hydrocarbons Sectional Committee, PCD 9

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Including Aromatic Hydrocarbons Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1972. Since then lot of changes have taken place in its technology and usage. Hence the Committee responsible for formulation of this standard decided to revise the standard to update it and align it with the international standards.

A new requirement of purity and related impurities by GLC method has been incorporated besides requirement of moisture in this revision.

o-Dichlorobenzene is primarily used as solvent and as a cleaning agent. It is particularly useful for removing grease, tar and resins. It is also being used in the synthesis of some organic chemicals.

In high concentration *o*-Dichlorobenzene vapour is irritating to the eyes and upper respiratory system. It has a slight degree of toxicity. Prolonged and repeated skin contact may be irritating and should be avoided. *o*-Dichlorobenzene is combustible and in case of fire involving it, water can be used successfully if applied in the form of a fog or spray. Carbon dioxide and dry chemical extinguishers are effective on small fires.

Following International Standard exist on the subject:

ISO 1698 : 1977 '*o*-Dichlorobenzene for industrial use — List of methods of test'

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

o-DICHLOROBENZENE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for *o*-Dichlorobenzene, technical for industrial use.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2362 : 1993	Determination of water by the Karl Fischer method (*first revision*)
4730 : 1994	Methods for determination of density of liquids (*first revision*)
4905 : 1968	Method for random sampling
5298 : 1983	Method for determination of distillation range and of distillation yield (*first revision*)
5813 : 1970	Method for determination of crystallizing point
9190 : 1979	Methods for determination of acidity and alkalinity in halogenated organic solvents and their admixture

3 REQUIREMENTS

3.1 Description

The material shall be clear, colourless to pale straw colour liquid free from suspended matter.

3.2 The material shall comply with the requirements prescribed in Table 1.

4 PRECAUTIONS IN STORING AND HANDLING

o-Dichlorobenzene is combustible and will burn if it comes in contact with an open flame and therefore should not be stored near highly flammable and dangerously reactive materials. Open flames and smoking shall not be allowed where it is stored or handled (*see also* Foreword and **5.2.1**).

5 PACKING AND MARKING

5.1 The material shall be packed, stored and transported in galvanized or mild steel drums. All the containers in which the material is packed shall be dry, clean and free from substances soluble in *o*-Dichlorobenzene.

5.2 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

5.2.1 The containers shall be marked with the following words in capitals 'CAUTION : VAPOUR IRRITATING'.

5.2.2 Each container shall be marked with the following:

a) Name of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Batch number and/or date of manufacture; and
d) Net mass of the contents.

5.2.3 *BIS Certification Marking*

The container may also be marked with the Standard Mark.

5.2.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn and their conformity determined as prescribed in Annex E.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for *o*-Dichlorobenzene, Technical
(*Clause* 3.2)

Sl No.	Characteristics	Requirements	Method of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i)	Relative density at 27/27°C	1.290 to 1.305	–	4730
ii)	Distillation yield, the temperature being corrected for a pressure of 760 mm Hg	a) Not more than 5 percent by volume below 176°C	–	5298
		b) 90 percent by volume, minimum between 176 and 182°C	–	–
iii)	Crystallizing point, *Max*	-5°C	A	–
iv)	Residue on evaporation, percent by mass, *Max*	0.025	B	–
v)	Acidity, ppm, *Max*	30	–	**4.2.2.** of 9190
vi)	Free chlorine	To pass the test	C	–
vii)	Moisture, percent by mass, *Max*	0.01	–	2362
viii)	Purity, percent by mass, *Max*	98.0	D	–
ix)	Benzene, percent by mass, *Max*	1.0	D	–
x)	Other isomers, percent by mass, *Max*	0.5	D	–

NOTE — The correction factor for relative density is +0.001 for each degree Celsius fall in temperature and –0.001 for each degree Celsius rise in temperature within the range 25-35°C.

ANNEX A

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF CRYSTALLIZING POINT

A-1 PROCEDURE

Determine the crystallizing point by the procedure as prescribed in IS 5813.

ANNEX B

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF RESIDUE ON EVAPORATION

B-1 APPARATUS

B-1.1 Basin — porcelain or silica.

B-2 PROCEDURE

Evaporate 50 ml of the material, accurately weighed in the tared basin over a steam-bath till dryness. Wipe the bottom and dry the residue in an air-oven at 180 ± 5 °C for 2h. Cool in a desiccator and weigh.

B-3 CALCULATION

$$\text{Residue on evaporation, percent by mass} = \frac{A-B}{C-B} \times 100$$

where

A = mass in g of the basin with the residue,

B = mass in g of the basin, and

C = mass in g of the basin with the material.

ANNEX C

[*Table* 1, *Sl No.* (vi)]

TEST FOR FREE CHLORINE

C-1 REAGENTS

C-1.1 Glacial Acetic Acid

C-1.2 3,3'-Dimethylnaphthidine Solution

Dissolve 0.01 g of finely ground 3, 3'-dimethylnaphthidine in 5 ml of glacial acetic acid and dilute rapidly to 200 ml. Store the solution in the dark.

C-2 PROCEDURE

To 50 ml of the material contained in a measuring cylinder, add 5 ml of the 3, 3' dimethylnaphthidine solution and shake the cylinder for 30 s.

C-2.1 The material shall be regarded to have passed the test if no pink colour is developed.

ANNEX D

[*Table* 1 *Sl No.* (viii), (ix) *and* (x)]

DETERMINATION OF PURITY BY GLC METHOD

D-1 OUTLINE OF THE METHOD

A small amount of the material is introduced into a gas liquid partition column. The various components that are volatile under the conditions of test are vapourized and transported through the column by a carrier gas. The separated components are measured in the effluent by a detector and recorded as a chromatogram. The chromatogram is interpreted by applying component attenuation and detector response factors to the peak areas and the relative concentrations are determined by relating the individual peak responses to the total peak responses. A typical chromatogram has been given in Fig. 1 for guidance only.

D-2 APPARATUS

Any gas chromatograph equipped with FID (Flame Ionization Detector) and electronic integrator may be used. The chromatographic conditions given here are for guidance only.

Column	:	15 percent OV-101 on chromosorb W (AW) 80-100 mesh
Length × O.D	:	3m × 4 mm — Glass
Temperature	:	Column oven 130°C
Injection port		225°C
Detector		225°C
Flow rate	:	Carrier gas nitrogen (N_2) 20 ml/*Min* H_2 30 ml/*Min* Air 300 ml/*Min*
Chart speed	:	20 mm/*Min*

D-3 REAGENTS

a) Benzene A.R;
b) A standard reference material of *o*-Dichlorobenzene of known purity; and
c) Sample of *o*-Dichlorobenzene with *m*-Dichlorobenzene, *p*-Dichlorobenzene, monochlorobenzene and benzene as impurities of known concentration.

D-4 PROCEDURE

Set the gas chromatograph. Inject 0.2 µl of sample with the help of 5 µl syringe. Allow chromatograph to develop. At the end of the run find out area percentage of all the components.

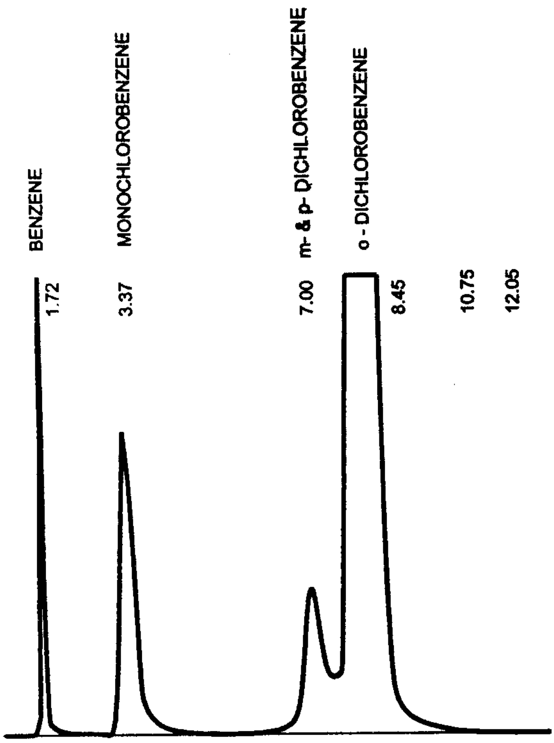

FIG. 1 A TYPICAL CHROMATOGRAM OF DICHLOROBENZENE

A standard blend containing necessary impurities may be injected into chromatographic system to verify the impurities present in product.

D-5 CALCULATIONS

Peak areas are calculated either by the most commonly used triangular method or automated integration. The sum of the areas is equated to 100 percent, it is implied that all substances have come out of the column and all substances have the same response factor, however, this is not the case in truth due to response factor of detector. Yet this method of calculation is fairly accurate.

The integrator automatically calculates the area percent of each individual impurity as follows:

$$\text{Area percent of '}X\text{'} = \frac{A \times 100}{\text{Area of all peaks}}$$

where

A = area of component 'X'.

Area percent of each impurity when multiplied with response factor will give the actual mass percent of the concerned impurity.

percent 'X' = Area percent of 'X' × Response factor

Assay as *o*-Dichlorobenzene, percent by mass = 100 – Sum of the all impurity percent.

Therefore

Purity (Assay) of *o*-Dichlorobenzene = 100 – [Water + Total area percent impurities].

ANNEX E

(*Clause* 6)

SAMPLING OF *o*-DICHLOROBENZENE

E-1 GENERAL REQUIREMENTS

E-1.1 In drawing, preparing, storing and handling test samples, the precautions mentioned in **4.1** and **5.1** shall be strictly observed.

E-1.2 Sampling instruments and containers shall be kept clean and dry.

E-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

E-1.4 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture and other important particulars of the consignment.

E-2 SAMPLING INSTRUMENT

E-2.1 To take out samples of *o*-Dichlorobenzene from various depths in large tanks, sampling bottle or can, as described below, may be used.

E-2.1.1 *Sampling Bottle or Can*

It shall consist of a weighed bottle or metal container with removable stopper or top, to which is attached a light chain. The bottle or can is fastened to a suitable pole. For taking sample, it is lowered in the drum or tank to the required depth and the stopper is removed by means of a chain for filling the container.

E-2.2 For small containers, sampling tube of suitable size may be used.

E-3 SCALE OF SAMPLING

E-3.1 Lot

In any consignment all the container of the same size, containing material of the same grade and drawn from the same batch of manufacture shall constitute a lot.

E-3.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this standard. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

E-3.3 The containers shall be chosen at random from the lot with the help of a suitable random number table. Reference may be made to IS 4905 for guidance to random selection procedures.

Table 2 Scale of Sampling

(*Clause* E-3.2)

Sl No.	Lot Size *n*	No. of Containers to be Selected *N*
(1)	(2)	(3)
i)	Up to 15	3
ii)	16 to 40	4
iii)	41 to 65	5
iv)	66 to 110	7
v)	111 and above	10

NOTE — Where the size of the lot is three or less, all the containers shall be sampled.

E-4 TEST SAMPLE AND REFEREE SAMPLE

E-4.1 From each of the containers selected as in **E-3.2**, draw with the help of a sampling bottle a representative portion of the material from different part of the container. Out of this portion from each container equal quantity of the material shall be taken and thoroughly mixed together to form a composite sample of about 1 500 ml in volume. This composite sample shall be thoroughly mixed and divided into three equal portions, one for the purchase, another for the supplier and the third for the referee.

E-4.2 The remaining portion corresponding to each of the selected containers (*see* **E-3.2**) shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *N* containers selected shall be for the purchaser, another for the supplier and the third for the referee.

E-4.3 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **E-1.4**.

E-4.4 The referee samples consisting of a composite sample and a set of *n* individual samples, shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

E-5 TESTS

E-5.1 Tests for description and crystallizing point shall be conducted on individual samples.

E-5.2 Tests for the remaining characteristics shall be conducted on the composite samples.

E-6 CRITERIA FOR CONFORMITY

E-6.1 For Individual Samples

The lot shall be declared as conforming to the requirements of description and crystallizing point if each of the test results of the individual samples satisfies the corresponding requirements of the test.

E-6.2 Composite Sample

For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results shall satisfy the relevant requirements given in Table 1.

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